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Wagner, Jakob Birkedal

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Stimulated Dynamic Electron Microscopy

Jakob B. Wagner

Center for Electron Nanoscopy, Technical University of Denmark, DK-2800 Kgs. Lyngby, Denmark

Monitoring the dynamic nature of nanostructured materials under various stimuli offers the opportunity for studying the dynamic structure-functionality relationship leading to insight and understanding of relevant phenomena for tailor-made materials.

Electron microscopy combined with various stimuli, that being gas exposure, heating, light, biasing, indentation, etc. is a unique tool for such monitoring. Both structural and compositional characterization of materials in active environments are becoming more and more accessible throughout laboratories around the Globe.

Here, a few examples on how stimulated dynamic electron microscopy has shed light on the nature of the nanomaterial dynamics, are discussed - mainly focusing on gas exposure, heat, and visible light treatment.

The redox behavior of nanostructured nickel is of great importance in a variety of applications, including solid oxide fuel cells. Here, the Ni serves as an active part of the anode placed on the fuel side of the cell in form of sub-micron sized grains co-sintered with a ceramic material. During operation at elevated temperature, failure and severe irreversible destruction of the anode will happen due to oxidation of the Ni if exposed to non-reducing conditions. In Fig. 1 the morphological and chemical development of the redox treatment of Ni(O) is shown. The initial NiO agglomerates are reduced in 130 Pa H_2 followed by an oxidation in 320 Pa O_2, both at 600˚C. Furthermore, co-sintered Ni-ceramic matrices are treated at similar conditions in the environmental transmission electron microscope (ETEM) in order to elucidate the possible effect of the ceramic on the redox behavior of Ni. [1,2]

Photocatalysts for solar fuel production are subject to intensive investigation as they constitute one viable route for solar energy harvesting. Cuprous oxide (Cu_2O) is a working photocatalyst for hydrogen evolution but it photocorrodes upon light illumination in an aqueous environment. In Fig. 2 the morphological and chemical evolution of Cu_2O cubes are followed by means of ETEM. The presence of H_2O (500 Pa) and visible light (405 nm) results in development of metallic copper grains in the cubes as confirmed by electron energy-loss spectroscopy. The water-light treatment are performed with the electron beam blanked due to electron beam effects. [3]

A few other examples will serve as a teaser for the vast opportunities stimulated electron microscopy in general and environmental transmission electron microscopy in particular gives researchers within the field of energy conversion processes.

[1] Q. Jeangros, A. Faes, J.B. Wagner, T.W. Hansen, U. Aschauer, J. Van herle, A. Hessler-Wyser, R.E. Dunin-Borkowski, Acta Materialia 58 (2010) 4578.

[2] Q. Jeangros, T.W. Hansen, J.B. Wagner, Acta Materialia 67 (2014) 362.

[3] F. Cavalca, A.B. Laursen, J.B. Wagner, C.D. Damsgaard, I. Chorkendorff, T.W. Hansen, Chemcatchem 5 (2013) 2667.

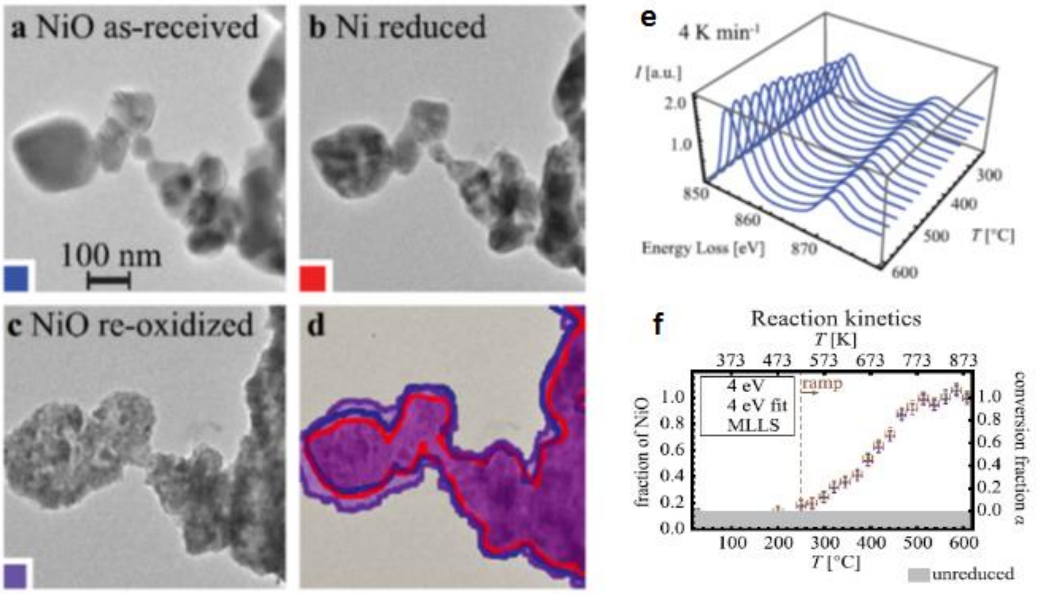

Fig 1: *Structural and compositional changes in a set of NiO particles (initial structure shown in (a)) observed after (b) reduction and (c) re-oxidation. The projected areas of (a–c) are superimposed in (d). e) Background-subtracted EEL spectra acquired as a function of temperature for a 4 K min*$^{-1}$ *ramp (normalized to unity at* L_2*). NiO and Ni experimental reference spectra were used to determine the oxidation kinetics (f).*

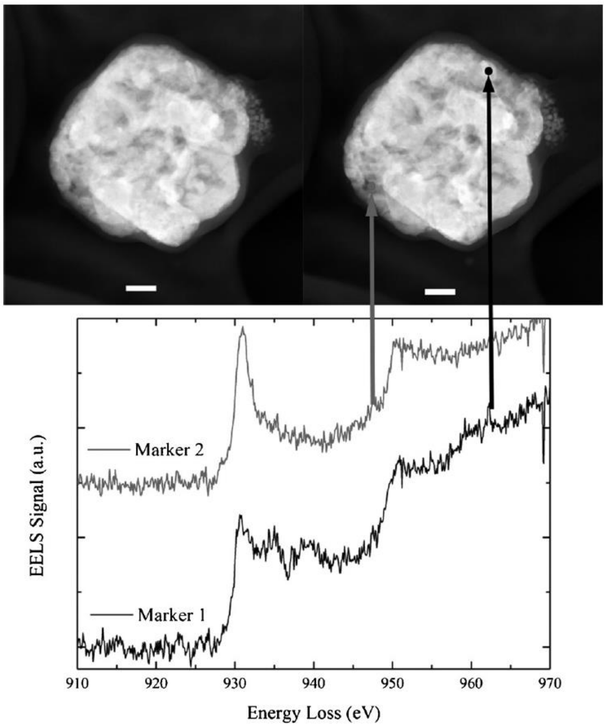

FIG 2: *HAADF-STEM images of a* Cu_2O *nanocube during exposure to* H_2O *and light. The images show how reduced crystals nucleate and grow within the particle body. STEM EELS on the marked positions is used to investigate the chemical phase of individual grains. The plot shows that a predominantly reduced phase at position 1 and an oxidized phase at position 2 coexist in the particle after 45 min exposure*. Scale bar 50nm.